

US-PAT-NO: 6077392

DOCUMENT-IDENTIFIER: US 6077392 A

TITLE: Text and cover printing paper and process for making the same

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INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE
Ruch; Thomas D.	Gansevoort	NY	N/A
N/A			

US-CL-CURRENT: 162/135,106/206.1 ,106/409 ,106/493 ,106/501.1  
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,428/220

DOCUMENT-IDENTIFIER: US 4094736 A  
TITLE: Preparation of cellulosic materials

ABPL:

The invention relates to a process for preparing paper or cardboard in which starch and a mineral filler are incorporated into cellulosic fibres. The process uses a blend, which is prepared under specified conditions, of raw starch and starch phosphate. The procedure for incorporating the starch blend and the mineral filler into the cellulosic fibres is also specified.

BSPR:

This invention concerns a filler material which is suitable for filling paper pulp and other cellulosic materials.

BSPR:

Mineral materials such as calcium carbonate, kaolin and titanium dioxide are well known as fillers for cellulosic materials. They can improve the opacity, whiteness and ink receptivity of paper in which they are contained but generally have a deleterious effect on the strength of the paper. Also, because the mineral materials are generally relatively cheap compared with the paper pulp the overall cost per unit weight of paper containing them is reduced. One problem associated with incorporating a mineral filler material in paper pulp is that the mineral particles must be relatively fine, i.e. of diameter about 50 microns or smaller, in order to confer the desirable improvements in opacity, whiteness and ink receptivity. If a mineral material has a particle size distribution such that substantially all of the particles are smaller than 50 microns there will generally be an appreciable proportion of particles having diameters of 1 or 2 microns or smaller. Many of these finer particles will not be retained in the mat of cellulosic

fibres which  
forms the paper with the result that some of the mineral filler  
material passes  
through the wire of the papermaking machine in the form of what  
is known in the  
paper-making art as "white water". It is generally difficult to  
recover  
mineral particles and cellulose fibres from white water but  
regulations  
regarding industrial effluents are widely being made more  
stringent so that  
foreign solid materials must be removed from effluent water  
before it is  
discharged to a river or stream.

BSPR:

An object of this invention is to provide an improved filler  
material for  
cellulosic products which confers the advantages of improved  
brightness,  
opacity and ink receptivity and which has less adverse effect on  
the strength  
of the paper as compared with a conventional filler, and is  
substantially  
completely retained in the mat of cellulosic fibres during the  
paper-making  
process.

BSPR:

According to the present invention, there is provided a process  
for preparing  
paper or cardboard which contains a filler composition, which  
process comprises  
(a) suspending raw starch in sufficient cold water to form a  
suspension  
containing about 3 to 10% by weight of starch solids; (b) heating  
the  
suspension thus obtained, with stirring, to a temperature in the  
range  
75.degree.-90.degree. C; (c) adding a starch phosphate to  
sufficient water so  
as to form a suspension or solution containing about 1-10% by  
weight of the  
starch phosphate; (d) adding the starch phosphate solution or  
suspension to the  
suspension of raw starch and raising the temperature of the  
mixture thus  
obtained to within the range of 75.degree.-95.degree. C. and  
holding its  
temperature at that level for about 5 to 10 minutes; (e)  
agitating the solution

obtained to a suspension of cellulosic fibres, coagulating the mixed starches by adding a solution of a salt having a multivalent cation and thereafter raising the pH of the suspension to above 5.5; and (h) forming the suspension of cellulosic fibres containing the mixture of mineral and coagulated mixed starches into sheet material.

CLPR:

12. A process for preparing paper or cardboard which contains a filler composition, which process comprises the steps of (a) suspending raw potato starch in sufficient cold water to form a suspension containing about 3 to 10% by weight of starch solids; (b) heating the suspension thus obtained, with stirring, to a temperature in the range 75.degree.-95.degree. C; (c) adding a starch phosphate having a degree of substitution in the range of from 0.02 to 0.1 to sufficient water so as to form a suspension or solution containing about 1-10% by weight of the starch phosphate; (d) adding the starch phosphate solution or suspension to the suspension of raw starch to form a starch mixture containing from 5% to 20% by weight of the starch phosphate and from 95% to 80% by weight of the raw starch and raising the temperature of the mixture thus obtained to within the range of 75.degree.-95.degree. C. and holding its temperature at that level for about 5 to 10 minutes; (e) agitating the solution of mixed starches thus obtained in a high shear mixer for from about 1 to 5 minutes and thereafter allowing the mixed starch solution to cool; (f) adding the cooled mixed starch solution to a dry powdered mineral filler material so that the resultant mixed suspension contains about 5-25% dry weight of the starch mixture and about 95-75% by weight of dry mineral; (g) adding the mixed suspension thus obtained to a suspension of cellulosic fibres, coagulating the mixed starches by adding a solution of aluminium

sulphate or calcium chloride and thereafter raising the pH of the suspension to a value in the range 5.8 to 6.5; and (h) forming the suspension of cellulosic fibres containing the mixture of mineral and coagulated mixed starches into sheet material.

CLPR:

13. A process for preparing paper or cardboard which contains a filler composition, which process comprises the steps of: (a) suspending raw starch in sufficient cold water to form a suspension containing about 3 to 10% by weight of starch solids; (b) heating the suspension thus obtained, with stirring, to a temperature in the range 75.degree.-95.degree. C; (c) adding a starch phosphate to sufficient water so as to form a suspension or solution containing about 1-10% by weight of the starch phosphate; (d) adding the starch phosphate solution or suspension to the suspension of raw starch and raising the temperature of the mixture thus obtained to within the range of 75.degree.-95.degree. C. and holding its temperature at that level for about 5 to 10 minutes; (e) agitating the solution of mixed starches thus obtained in a high shear mixer for from about 1 to 5 minutes and thereafter allowing the mixed starch solution to cool; (f) adding the cooled mixed starch solution to a dry powdered mineral filler material so that the resultant mixed suspension contains about 5-25% dry weight of the starch mixture and about 95-75% by weight of dry mineral; (g) treating the mixed suspension obtained after step (f) with a solution of a salt having a multivalent cation so as to coagulate the starch mixture, raising the pH of the resultant material to above 5.5 and thereafter adding the resultant suspension to a suspension of cellulosic fibres; and (h) forming the suspension of cellulosic fibres containing the mixture of mineral and coagulated mixed starches into sheet material.

CLPR:

24. A process for preparing paper or cardboard which contains a filler composition, which process comprises the steps of: (a) suspending raw potato starch in sufficient cold water to form a suspension containing about 3% to 10% by weight of starch solids; (b) heating the suspension thus obtained, with stirring, to a temperature in the range 75.degree.-95.degree. C; (c) adding a starch phosphate having a degree of substitution in the range of from 0.02 to 0.1 to sufficient water so as to form a suspension or solution containing about 1-10% by weight of the starch phosphate; (d) adding the starch phosphate solution or suspension to the suspension of raw starch to form a starch mixture containing from 5% to 20% by weight of the starch phosphate and from 95% to 80% by weight of the raw starch and raising the temperature of the mixture thus obtained to within the range of 75.degree.-95.degree. C. and holding its temperature at that level for about 5 to 10 minutes; (e) agitating the solution of mixed starches thus obtained in a high shear mixer for from about 1 to 5 minutes and thereafter allowing the mixed starch solution to cool; (f) adding the cooled mixed starch solution to a dry powdered mineral filler material so that the resultant mixed suspension contains about 5-25% dry weight of the starch mixture and about 95-75% by weight of dry mineral; (g) treating the mixed suspension obtained after step (f) with a solution of aluminium sulphate or calcium chloride so as to coagulate the starch mixture, raising the pH of the resultant material to a value in the range 5.8 to 6.5 and thereafter adding the resultant suspension to a suspension of cellulosic fibres; and (h) forming the suspension of cellulosic fibres containing the mixture of mineral and coagulated mixed starches into sheet material.

DOCUMENT-IDENTIFIER: US 3798047 A  
TITLE: PIGMENT FOR COATING PAPER AND MANUFACTURE THEREOF

TTL:  
PIGMENT FOR COATING PAPER AND MANUFACTURE THEREOF

ABPL:  
A pigment for coating paper comprises an intimate mixture of 40-95 parts of natural calcium carbonate and 60-5 parts of satin white, at least 80 percent of the satin white being smaller than two microns. The pigment is prepared by grinding the components together, one of the components being in suspension.

BSPR:  
The present invention relates to improvements in white pigments for coating paper and the manufacture thereof.

BSPR:  
The most widely used paper coating pigment presently is kaolin because this pigment best meets the usual requirements for such pigments, which are excellent whiteness, great fineness (about 80 percent of the pigment particles being under two microns), great covering power or opacity, and an ability to acquire a shine on calendering.

BSPR:  
However, the requirements for printing coated paper are becoming constantly more severe. More particularly, it is desirable to increase the printing speed while obtaining sharp and brilliant printed images, as well as to obtain coated paper useful for offset printing, i.e., a paper which has a good water resistance as well as a high speed of ink absorption.

BSPR:  
It has also been proposed to use other pigments for coating paper, such as natural or precipitated calcium carbonates, which modify the structure of the

coating and which thus permit the micro-porosity of the coating to be adjusted.  
Despite their whiteness and high covering power or opacity, however, precipitated calcium carbonates have been used to a limited extent only because of the high viscosity of their aqueous suspensions. As to the natural calcium carbonates, while they are cheaper than kaolin and precipitated calcium carbonates, their use has been limited because they are not white enough and they do not develop a shine on calendering.

BSPR:

It is the primary object of this invention to overcome the disadvantages of the known paper coating pigments and to provide such a pigment which gives prints of good quality while being relatively inexpensive.

BSPR:

Coatings of the pigment of this invention, constituted by an interlacing of fine needles of satin white crystals with almost spherical particles of calcium carbonate have a perfectly smooth surface, which facilitates rotogravure impressions, and an optimum micro-porosity. The physical structure of these coatings is superior to the lamellar structure of kaolin coatings while being at least equal thereto in gloss, brightness, smoothness and opacity. Therefore, these coatings give better printing properties.

DEPR:

The pigment was applied in a thin coating on a black surface and its brightness was compared with that obtained with the same thickness of a kaolin coating. The respective brightness, measured by an Elrepho apparatus equipped with a blue filter which passes light having a wavelength between 4,300 and 4,600 Å, were as follows:

DEPR:

Thus, the pigment, which contained about 85 percent, by weight, of calcium carbonate and about 15 percent, by weight, of satin white, had a



whiteness  
superior to that of the conventional kaolin coating. This  
pigment, while  
useful in other applications, is particularly advantageous for  
paper coatings  
since it also has excellent absorptivity for ink and, in  
suspension, has a  
viscosity lower than that of a kaolin suspension of the same  
concentration.

DEPR:

Tests showed the resultant product to have a fineness and  
covering power or  
opacity superior to that of satin white obtained by the reaction  
of aluminum  
sulfate and lime.

DEPR:

In the same brightness test as in Example 1, the following  
results were  
obtained:

DEPR:

Thus, the directly produced pigment proved to have the best  
brightness  
characteristics. In addition, the direct method is particularly  
simple, and  
the use of  $\text{CaCO}_3$  instead of lime produces considerable  
economies in  
manufacture.

CLPR:

1. A process of preparing a pigment for coating paper,  
comprising the steps of  
preparing a suspension of satin white and grinding the suspension  
of satin  
white with natural calcium carbonate until at least about 80  
percent, by  
weight, of the resulting pigment has a particle size of less than  
about two  
microns, the amount of satin white and calcium carbonate being  
such that the  
resultant intimate mixture thereof comprises about 95 to 40  
parts, by weight,  
of calcium carbonate and 5 to 60 parts, by weight, of satin  
white.

CLPR:

5. A process of preparing a pigment for coating paper,  
comprising the steps of  
grinding natural calcium carbonate in an aqueous suspension with

an amount of  
aluminum sulfate sufficient to react only with a portion of the  
calcium  
carbonate to produce satin white until an intimate mixture of the  
natural  
calcium carbonate and satin white is obtained and at least 80  
percent, by  
weight, of the resulting pigment has a particle size of less than  
about two  
microns, the amounts of calcium carbonate and aluminum sulfate  
being such that  
the intimate ground mixture thereof comprises about 95 to 40  
parts, by weight,  
of calcium carbonate and 5 to 60 parts, by weight, of satin  
white.

of mixed starches thus obtained in a high shear mixer for from about 1 to 5 minutes and thereafter allowing the mixed starch solution to cool; (f) adding the cooled mixed starch solution to a dry powdered mineral filler material so that the resultant mixed suspension contains about 5-25% dry weight of the starch mixture and about 95-75% by weight of dry mineral; (g) either adding the mixed suspension thus obtained to a suspension of cellulosic fibres, coagulating the mixed starches by adding a solution of a salt having a multivalent cation and thereafter raising the pH of the suspension to above 5.5; or treating the mixed suspension obtained after step (f) with a solution of a salt having a multivalent cation so as to coagulate the starch mixture, raising the pH of the resultant material to above 5.5 and thereafter adding the resultant suspension to a suspension of cellulosic fibres; and (h) forming the suspension of cellulosic fibres containing the mixture of mineral and coagulated mixed starches into sheet material.

**BSPR:**

The mechanical working of step (e) improves the behaviour of the starch mixture. The two types of polymer become entangled as a result of the high shear mixing, and this tends to improve the homogeneity of coagulation subsequently effected, as well as improving the association between the mixed starches and the mineral filler particles. Because of the improvement in coagulation effected thereby, the amount of starch which remains in solution is reduced; this is advantageous, because dissolved starch in water ultimately recovered from, for example, the paper making process causes difficulties in the reuse of the water.

**DEPR:**

Hand sheets of paper filled with starch/clay agglomerates in accordance with the invention were prepared in the following way.

DEPR:

400 g. of bleached sulphite spruce pulp were soaked in 10 liters of water for 4 hours and the mixture was then disintegrated for 10 minutes in a turbine mixer manufactured by Etablissements Cellier of Aix-les-Bains, France, the impeller rotating at a speed of 220 r.p.m. The contents of the mixer were washed out with a further 10 liters of water and transferred to a laboratory beater where a further 2 liters of water were added and the mixture beaten for 161/2 minutes. At this stage the stock contained approximately 1.8% by weight of dry pulp. The beating time was chosen to give the optimum compromise between brightness and strength properties of the stock; this was such that the beaten pulp had a Canadian Standard Freeness of about 300-320 cc. (This is measured by placing a 1 liter sample of an aqueous suspension containing 0.3% by weight of fibres in a container provided with a wire mesh base covered with a water tight hinged lid. The hinged lid is swung open to allow water draining through the pulp and through the wire mesh to fall into a large funnel having a narrow orifice at the base of its central stem and an overflow pipe a given height above the bottom orifice. The freer the pulp, the more rapidly water falls into the funnel, and the more water overflows down the pipe provided because the orifice is too narrow to accommodate the total flow of water. The volume of water overflowing is measured in a measuring cylinder and the Canadian Standard Freeness is expressed in cubic centimeters of water). The following table gives the "single sheet brightness" (percentage reflectance of violet light of wavelength 458 nm. from the surface of a single sheet of paper having a dry weight of 60 grams per square meter) and the burst ratio for paper sheets formed from a bleached sulphite softwood pulp beaten in a Valley "Niagara" beater to different Canadian Standard Freeness values:

DEPR:

800 ml. of the stock were then made up to 2 liters with water and disintegrated in a laboratory disintegrator which was operated for 15,000 revolutions of the impeller. The volume of stock was made up to 4 liters and the consistency was checked by filtering and evaporating to dryness a small sample and weighing the residue. Water was added if necessary to reduce the consistency to 0.3% by weight of dry pulp. Filler was added and stirred in by hand at the rate of 14.5 g of filler per 4 liters of paper fibre stock.

DEPR:

Seven 14.5 g. batches of starch/clay mixture were prepared as in Example 1 and each batch was treated with a different quantity of a solution of aluminium sulphate containing 5 g. of  $\text{Al}_2(\text{SO}_4)_3 \cdot 16\text{H}_2\text{O}$  per 100 ml. of solution. The pH was adjusted to 6.0 with sodium hydroxide, all mixing being performed by hand so that the reaction product of clay with the starch was subjected only to very small shearing forces. The clay with its coating of coagulated starch in the form of sliver-shaped agglomerates was then mixed with 4 liters of paper pulp stock and formed into hand sheets as described in Example 2 above. The hand sheets from each batch were tested for bursting strength by the test prescribed in TAPPI Standard No. T 403 os-74. The bursting strength is defined as the hydrostatic pressure in kilonewtons per square meter required to produce rupture of the material when the pressure is increased at a controlled constant rate through a rubber diaphragm to a circular area 30.5 mm in diameter. The area of the material under test is initially flat and held rigidly at the circumference but free to bulge during the test. Samples of each sheet were also weighed dry and then incinerated, the weight of the dry sample being used to determine the weight

per unit area  
of the paper in grams per square meter and the weight of ash to  
calculate the  
percentage of filler material (clay and starch) based on the  
weight of dry  
fibres after allowing for the loss on ignition of the filler and  
also to  
calculate the percentage by weight of the added filler material  
which was  
actually retained by the fibres.

DEPR:

The burst strengths were divided by the weight per unit area of  
the paper to  
give a burst ratio and the burst ratio for each sheet of filled  
paper was then  
expressed as a percentage of the burst ratio for a sheet of paper  
prepared from  
the same stock but containing no filler.

DEPR:

As a comparison, hand sheets were also prepared from the same  
paper stock but  
containing as the filler only the English china clay described in  
Example 1.  
Burst ratios and percentages by weight of inorganic filler  
material were  
determined for two different filler loadings.

DEPR:

These results show that, for a given loading of filler, the  
strength of the  
paper is reduced less by the addition of china clay treated with  
a mixture of  
potato starch and starch phosphate coagulated by aluminium  
sulphate in  
accordance with the invention than by the addition of untreated  
china clay.  
However sufficient aluminium sulphate, i.e. at least 0.5 ml of 5%  
solution per  
14.5 g. of starch/clay mix or about 0.17% by weight based on the  
total weight  
of filler, must be added in order to coagulate the starch and  
starch phosphate  
fully and thus achieve good retention of the filler and a good  
improvement in  
strength over that obtained with a similar quantity of untreated  
china clay.  
These results may be compared with the results hitherto  
obtainable as  
exemplified by U.S. Pat. No. 3,132,066. The first three

entries in following  
Table III are derived from Example 23 of this U.S.  
Specification, while the  
remaining entries are derived from Table II above:

DEPR:

The above results show that when titanium dioxide is treated with starch phosphate in accordance with U.S. Pat. No. 3,132,066 the retention of titanium dioxide is unaffected. However when china clay is treated with a mixture of potato starch and starch phosphate coagulated by aluminium sulphate in accordance with the invention not only is the strength of the paper for a given loading of filler increased, but the retention of the filler is increased also.

DEPL:

It can be seen that increasing the beating time so as to reduce the freeness from 280 to 137 results in a small improvement in strength at the expense of a significant drop in brightness.

DEPL:

The results from U.S. Pat. No. 3,132,066 are not directly comparable with the results obtained by the process of this invention because the quantity of filler used, as well as the nature of the filler, are very different from those employed in the process of the invention. It is well known that if starch is added to paper pulp in quantity similar to that used in U.S. Pat. No. 3,132,066 the burst strength of the paper formed from the treated pulp is greater than that of paper formed from untreated pulp. It is also known that the effect of adding titanium oxide, or other mineral filler, to the pulp is to reduce the burst strength of the paper formed. However in the particular case described in U.S. Pat. No. 3,132,066 the amount of titanium dioxide added is very small so it is not surprising that the beneficial action of the starch more than counteracts the deleterious action of the titanium

dioxide. It is the aim of our invention to incorporate in the paper much greater quantities of filler without reducing the strength to an undesirable level and so produce a paper which has a higher proportion of the relatively inexpensive filler material and a smaller proportion of the expensive paper pulp.

DEPC:

Preparation of paper hand sheets

DETL:

Table I \_\_\_\_\_ Canadian Single Standard Sheet

Freeness	Brightness	Burst	(c.c.)	(%)	Ratio
280	59.3	47	642	70.5	18 468 65.0 35

137	55.3	50			
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CLPR:

1. A process for preparing paper or cardboard which contains a filler composition, which process comprises the steps of (a) suspending raw starch in sufficient cold water to form a suspension containing about 3 to 10% by weight of starch solids; (b) heating the suspension thus obtained, with stirring, to a temperature in the range 75.degree.-95.degree. C; (c) adding a starch phosphate to sufficient water so as to form a suspension or solution containing about 1-10% by weight of the starch phosphate; (d) adding the starch phosphate solution or suspension to the suspension of raw starch and raising the temperature of the mixture thus obtained to within the range of 75.degree.-95.degree. C. and holding its temperature at that level for about 5 to 10 minutes; (e) agitating the solution of mixed starches thus obtained in a high shear mixer for from about 1 to 5 minutes and thereafter allowing the mixed starch solution to cool; (f) adding the cooled mixed starch solution to a dry powdered mineral filler material so that the resultant mixed suspension contains about 5-25% dry weight of the starch mixture and about 95-75% by weight of dry mineral; (g) adding the mixed suspension thus



US-PAT-NO: 6105325

DOCUMENT-IDENTIFIER: US 6105325 A

TITLE: Method, assembly and additional coat  
for the construction of interior works

----- KWIC -----

The other sheet of plasterboards has a visible face, called a lining face, of a colour generally lighter than the grey sheet. To obtain this lighter colour, the layer or layers of this face are based on chemical pulp, if appropriately bleached, composed of recycled and/or new cellulose fibres, and/or on mechanical pulp, if appropriately bleached. By chemical pulp is meant a pulp obtained by eliminating a very large proportion of the non-cellulose components from the raw material by chemical treatment, for example, by cooking in the presence of suitable chemical agents, such as soda or bisulphites. When this chemical treatment is completed by bleaching, a large part of the coloured substances is eliminated, as well as the substances which risk decomposing by ageing and giving unpleasant yellow shades associated with the presence of, for example, lignin.

in a continuous centrifuge to remove oversize material followed by leaching to remove iron-based colored compounds. In the leaching process the kaolin is acidified with H<sub>2</sub>SO<sub>4</sub> to a pH of 3.0 to solubilize the iron. Sodium hydrosulfite is then added to reduce the iron to a more soluble ferrous form which is removed during the dewatering process. The flocculated clay, generally at approximately 30% solids by weight, is then filtered, such as by dewatering on a rotary vacuum filter to a solids level approximately 60% by weight. The filter cake is then either dried or redispersed with additional dry clay if it is to be sold as approximately 70% by weight solids slurry. To produce high brightness products, i.e., a product having a brightness index greater than 90, impurities may be removed from the kaolin clay by further processing the kaolin clay through flotation or magnetic separation. To produce a delaminated product, the coarse fraction from the initial centrifugation is ground in sand grinders to shear the stacks of platelets normally found in kaolin and thereby produce individual particles having an equivalent spherical diameter less than 2 microns.

#### BSPR:

It is well appreciated in the art that kaolin clay pigments must have certain rheological and optical properties to be suitable for use in paper manufacture as paper coatings or paper fillers. The kaolin clay pigment must be available as a high solids suspension typically having a clay solids content of about 50% to about 70% by weight, but still exhibiting a viscosity low enough to permit efficient and economical pumping, mixability with other filler or coating components, and application to the paper. Additionally, it is of utmost importance that the kaolin pigment exhibit certain optical properties, namely high brightness, high gloss and high opacity.

BSPR:

The influence of particle size distribution upon the optical properties of kaolin pigments has long been appreciated in the art. For example, in commonly assigned U.S. Pat. No. 2,992,936, Rowland discloses that a kaolin clay product having the following particle size distribution (in terms of equivalent spherical diameter, e.s.d.) will consistently show improved brightness, gloss and opacity when used as a paper coating clay:

BSPR:

In a paper entitled "Chemically Induced Kaolin Floc Structures for Improved Paper Coating", presented at the 1983 TAPPI Coating Conference, W. H. Bundy et al. disclosed an improved high bulking paper coating pigment, referred to as 1089, which comprises a chemically modified kaolin produced by the Georgia Kaolin Company, Inc. and marketed under the trade name Astra-Lite. Structures of optimum functionality are said to be derived by chemically treating a base kaolin clay having a particle size distribution wherein from about 80% to 93% by weight of the kaolin particles are less than 2 microns e.s.d. to selectively flocculate a portion of the submicron fines therein thereby aggregating a portion of these fines on the surface of larger kaolin platelets and effectively inactivating a large portion of colloidal particles. Such a chemically modified kaolin coating pigment derived from a base kaolin wherein 92% by weight particles under 2 microns is presented by Bundy et al. as having a particle size distribution as follows:

BSPR:

There is disclosed in U.S. Pat. No. 4,738,726, an opacifying pigment composition suitable for use as a paper filler or coating which consists essentially of particles of hydrous kaolin clay flocculated with a controlled minor amount of a cationic polyelectrolytic flocculent, e.g., a

quaternary  
ammonium polymer salt or a diallyl ammonium polymer salt. The  
base kaolin clay  
is selected to have a particle size distribution prior to  
flocculation wherein  
less than 35% by weight are finer than 0.3 microns, i.e.,  
colloidal.

DEPR:

All surface areas herein referred to are determined by a slightly  
modified  
version of the standard methylene blue spot test procedure  
outlined by M. J.  
Nevins and D. J. Weintritt in an article entitled "Determination  
of Cation  
Exchange Capacity by Methylene Blue Adsorption", published in the  
American  
Ceramics Society Bulletin, Volume 46, pages 587-592, 1967. In  
accordance with  
this procedure, a one gram sample of powdered kaolin clay  
prepared by spray  
drying and pulverizing the underflow kaolin slurry produced after  
defining via  
controlled centrifugation was weighed into a 100 ml. beaker.  
Ten milliliters  
of deionized water was then added and the aqueous clay suspension  
in the beaker  
continuously agitated with a magnetic stirrer and stirring bar  
while methylene  
blue stock solution was added in 0.5 milliliter increments.  
Approximately one  
minute after each addition of methylene blue, a drop of liquid  
was removed from  
the beaker with a glass rod and deposited on a piece of Whatman  
#50 filter  
paper. The addition of methylene blue ceased when the dye first  
appeared as a  
blue ring surrounding the dyed solids formed on the filter paper,  
the blue ring  
indicating unabsorbed dye. The suspension was then mixed for an  
additional two  
minutes after the initial appearance of the blue ring to assure  
maximum  
adsorption of the dye by the clay solids in the suspension. An  
additional drop  
was then removed from the beaker and placed on the filter paper. .  
If the blue  
ring again appeared, the test was considered completed. If not,  
the addition  
on methylene blue stock was continued in 0.5 milliliter  
increments until a

stable blue ring was obtained.

DEPR:

The underflow aqueous kaolin clay suspension 35 from the centrifugation step is collected as the desired product and further processed according to its intended use. For example, if the product is to be used as a paper coating clay, the underflow aqueous kaolin clay suspension 35 from the centrifugation step, which is typically at a solids content of about 40% to about 50% by weight, is first diluted with water to a solids content of about 15% to about 25% solids by weight, treated with sulfuric acid to reduce its pH to a level between 2.5 and 3.0, and leached in a conventional manner by adding thereto an aqueous solution of a reducing agent, for example sodium dithionite solution at a treatment level of 2 to 6 pounds of sodium dithionite per ton of dry clay. After leaching to improve brightness, the pH of the leached aqueous kaolin clay suspension is adjusted to 3.0, filtered on a rotary vacuum filter, rinsed and reblunged. A portion of this suspension is then spray dried and the spray dried product pulverized and remixed with the remainder of the suspension to produce the desired coating clay product at a solids level about 65% solids by weight.

DEPR:

If, however, the underflow aqueous kaolin clay suspension 35 from the centrifugation step is to be used for paper filling applications, the underflow aqueous kaolin clay suspension 35 is again first diluted with water to a solids content of about 15% to about 25% solids by weight, but thence prior to leaching is treated with an amine, typically hexamethylenediamine at a treatment level of about 0.5 to about 2.5 pounds hexamethylenediamine per ton of dry clay, before adjusting the pH of the treated suspension to a level between 2.5 and 3.0, thence preferably adding aluminum sulfate

(alum) to the amine treated suspension, typically at a rate of about 10 to about 20 pounds of alum per ton of dry clay, after leaching and further processing the aqueous clay suspension as hereinbefore described with respect to the production of a coating clay product.

DEPR:

A comparison of the opacity improvements presented in Table I also confirms that defining by the two-step process of the present invention (Examples IV and V) wherein the underflow suspension, i.e., the coarse cut, from the first centrifugation step is subjected to a second centrifugation step and the overflow suspensions from both centrifugation steps, yields a defined product which exhibits a substantially greater opacity improvement when compared to a product defined at the same defining level but defined by the typical prior art two-step centrifugation exemplified by Example I wherein the overflow suspension, i.e., the finer cut, from the first centrifugation step is subjected to a second centrifugation step and the underflow suspensions from both the first and second centrifugation steps combined to yield the defined product as described in U.S. Pat. No. 2,992,936.

DEPR:

The treated and untreated products prepared as presented in Example VIII where used in formulate paper coating compositions by admixing 100 parts by weight of the sample product, 12 parts by weight of a latex binder, 6 parts by weight of a starch binder, and 1 part by weight of calcium stearate. For comparison purposes, coating compositions were also prepared via this formulation using a No. 1 coating clay and a No. 1 high brightness kaolin. Each coating formulation was then applied to a 47 lb/3300 ft.<sup>2</sup> basestock paper at a coating weight of 7.1 lb/3300 ft.<sup>2</sup>. The coated sheets were then calendered

two nips at 140.degree. and 200 psi pressure, before measurements of opacity, brightness, paper gloss and print gloss were taken in accordance with standard TAPPI methods, which measurements are reported in Table II.

DEPR:

A comparison of the opacity, brightness, the paper gloss and print gloss of the paper coatings prepared using the defined kaolin products of Example VIII, both treated and untreated, with those of paper coatings prepared using standard No. 1 coating clay or No. 1 high brightness clay clearly confirms that a defined kaolin clay produced not only by defining at low solids level in accordance with the present invention, but also prepared prior to defining by mechanically dispersing the aqueous kaolin clay via scrub grinding to break up kaolin agglomerates prior to chemically dispersing the suspension to minimum viscosity before defining, will exhibit superior performance as a coating clay with or without subsequent surface treatment.

DETL:

TABLE II _____ Paper Print								
Clay in Coating								
Opacity	Brightness	Gloss	Gloss					
					No. 1			
High Brightness	87.6	79.3	59	79	No. 1 Coating	87.7	78.2	58 79
Ex. VIII -								
Treated	88.8	80.7	69	88	Ex. VIII - Untreated	88.7	80.6	67 86

DERWENT-ACC-NO: 1999-217091  
DERWENT-WEEK: 200046  
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TITLE: Paper web production for making coated fine papers

INVENTOR: LESKELAE, M; NYGARD, S ; PITKAENEN, M

PATENT-ASSIGNEE: METSAE-SERLA OYJ[METSN], METSAE-SERLA  
OY[METSN]

PRIORITY-DATA: 1997FI-0003704 (September 16, 1997)

PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE	
PAGES	MAIN-IPC		
JP 3085935 B2	September 11, 2000	N/A	007
D21H 011/10			
EP 908557 A1	April 14, 1999	E	011
D21H 011/10			
FI 9703704 A	March 17, 1999	N/A	000
D21B 000/00			
FI 103417 B1	June 30, 1999	N/A	000
D21B 001/00			
CA 2247307 A1	March 16, 1999	E	000
D21H 011/10			
JP 11189983 A	July 13, 1999	N/A	023
N/A			

DESIGNATED-STATES: AL AT BE CH CY DE DK ES FI FR GB GR IE IT LI  
LT LU LV MC MK N  
L PT RO SE SI

APPLICATION-DATA:

PUB-NO	APPL-DESCRIPTOR	APPL-NO
APPL-DATE		
JP 3085935B2	N/A	1998JP-0262004
September 16, 1998		
JP 3085935B2	Previous Publ.	JP 11189983
N/A		
EP 908557A1	N/A	1998EP-0660093
September 16, 1998		
FI 9703704A	N/A	1997FI-0003704
September 16, 1997		
FI 103417B1	N/A	1997FI-0003704
September 16, 1997		
FI 103417B1	Previous Publ.	FI 9703704
N/A		



CA 2247307A1 . N/A  
September 16, 1998  
JP 11189983A N/A  
September 16, 1998

1998CA-2247307

1998JP-0262004

INT-CL (IPC): D21B000/00; D21B001/00 ; D21H011/10 ;  
D21H019/72 ;  
D21H019/82

ABSTRACTED-PUB-NO: EP 908557A

BASIC-ABSTRACT: NOVELTY - A paper web is formed from a fibrous raw material

stock on a conventional papermaking machine. The stock contains 20-70 % by dry

weight of mechanical pulp from Poplar trees. The remainder of the stock is

70-30 % by weight of bleached chemical softwood pulp.

DETAILED DESCRIPTION - INDEPENDENT CLAIMS are also included for (1) a base

paper of which 30-60 wt% of its fibers are derived from a mechanical aspen

pulp, 70-40 wt% from chemical softwood pulp, its grammage is 30-200 g/m<sup>2</sup>,

1.2-1.6 cm<sup>3</sup>/g and opacity and brightness over 78%; and (2) fine paper produced

from double-coated base paper, in which at least one of the coating layers is

formed from a coating color containing pigment with a steep particle size distribution.

USE - To provide a base paper web suitable for use in making coated fine papers.

ADVANTAGE - Addition of mechanical pulp as pressure groundwood pulp from Poplar trees with short fibers increases the bulk and light scattering of the paper.

CHOSEN-DRAWING: Dwg.0/0

TITLE-TERMS:

PAPER WEB PRODUCE COATING FINE PAPER

DERWENT-CLASS: F09

CPI-CODES: F05-A06;

UNLINKED-DERWENT-REGISTRY-NUMBERS: 1278U

SECONDARY-ACC-NO:

CPI Secondary Accession Numbers: C1999-064122

DERWENT-ACC-NO: 1996-161666  
DERWENT-WEEK: 199819  
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TITLE: Improved optical properties of paper i.e. increased  
brightness - by  
using as filler, blocky six sided rhombohedral calcium carbonate  
with crystal  
morphology, having specific surface area

INVENTOR: KUNESH, C J; PASSARETTI, J D

PATENT-ASSIGNEE: MINERALS TECHNOLOGIES INC[MINEN], PFIZER  
INCECHNOLOGIES  
INC[PFIZ]

PRIORITY-DATA: 1990US-0493075 (March 13, 1990)

PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE	
PAGES	MAIN-IPC		
ES 2112602 T3	April 1, 1998	N/A	000
C01F 011/18			
EP 703315 A2	March 27, 1996	E	025
D21H 017/67			
EP 703315 A3	June 26, 1996	N/A	000
N/A			
EP 703315 B1	January 14, 1998	E	026
C01F 011/18			
DE 69128730 E	February 19, 1998	N/A	000
C01F 011/18			

DESIGNATED-STATES: BE CH DE ES FR GB IT LI NL SE BE CH DE ES FR  
GB IT LI NL SE

CITED-DOCUMENTS: EP 10643; EP 179597 ; GB 540044 ; US 3320026

APPLICATION-DATA:

PUB-NO	APPL-DESCRIPTOR	APPL-NO
APPL-DATE		
ES 2112602T3	N/A	1995EP-0118362
March 5, 1991		
ES 2112602T3	Based on	EP 703315
N/A		
EP 703315A2	Div ex	1991EP-0301797
March 5, 1991		
EP 703315A2	N/A	1995EP-0118362
March 5, 1991		

EP 703315A3	Div ex	1991EP-0301797
March 5, 1991		
EP 703315A3	N/A	1995EP-0118362
March 5, 1991		
EP 703315B1	Div ex	1991EP-0301797
March 5, 1991		
EP 703315B1	N/A	1995EP-0118362
March 5, 1991		
EP 703315B1	Div ex	EP 447094
N/A		
DE69128730E	N/A	1991DE-0628730
March 5, 1991		
DE69128730E	N/A	1995EP-0118362
March 5, 1991		
DE69128730E	Based on	EP 703315
N/A		

INT-CL\_(IPC): C01F011/18; C09C000/00 ; D21H017/67

RELATED-ACC-NO: 1991-275702;1996-161576

ABSTRACTED-PUB-NO: EP 703315A

BASIC-ABSTRACT: Improved optical properties of paper made from calcium

carbonate having a blocky six-sided rhombohedral final crystal morphology, is

claimed. It has a surface area of 3 - 15 m<sup>2</sup>/g and an average discrete particle

size of 0.2 - 0.9  $\mu$ m. The discrete particles have an aspect ratio of less

than 2 and a particle size distribution such that at least 60 wt.% of the

particles lie within 50% of the equivalent discrete particle average spherical

dia.

USE - As a filler material in paper making.

ADVANTAGE - Improves the optical properties of paper i.e. increased brightness and opacity.

ABSTRACTED-PUB-NO: EP 703315B

EQUIVALENT-ABSTRACTS: Improved optical properties of paper made from calcium

carbonate having a blocky six-sided rhombohedral final crystal morphology, is

claimed. It has a surface area of 3 - 15 m<sup>2</sup>/g and an average discrete particle

size of 0.2 - 0.9  $\mu$ m. The discrete particles have an aspect ratio of less

than 2 and a particle size distribution such that at least 60 wt.% of the particles lie within 50% of the equivalent discrete particle average spherical dia.

USE - As a filler material in paper making.

ADVANTAGE - Improves the optical properties of paper i.e. increased brightness and opacity.

CHOSEN-DRAWING: Dwg.0/19 Dwg.0/19

TITLE-TERMS:

IMPROVE OPTICAL PROPERTIES PAPER INCREASE BRIGHT FILL SIX SIDE RHOMBOHEDRA L  
CALCIUM CARBONATE CRYSTAL MORPHOLOGY SPECIFIC SURFACE AREA

DERWENT-CLASS: E33 F09 G01

CPI-CODES: E34-D03; F05-A06D; G01-A01;

CHEMICAL-CODES:

Chemical Indexing M3 \*01\*

Fragmentation Code

A220 A940 C106 C108 C530 C730 C801 C802 C803 C805  
C807 M411 M781 M903 M904 M910 Q324 Q333 Q606 R032

Specific Compounds

01278U

Registry Numbers

1278U

UNLINKED-DERWENT-REGISTRY-NUMBERS: 1278U

SECONDARY-ACC-NO:

CPI Secondary Accession Numbers: C1996-051258

DERWENT-ACC-NO: 1990-254341  
DERWENT-WEEK: 199034  
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TITLE: High opacifying kaolin contg. pigments - useful as fillers in paper sheets

INVENTOR: DUNAWAY, W; TURNER, R E

PATENT-ASSIGNEE: ECC AMERICA INC[ENGC]

PRIORITY-DATA: 1988US-0288681 (December 22, 1988)

PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE	
PAGES	MAIN-IPC		
AU 8947058 A	June 28, 1990	N/A	000
N/A			
BR 8906697 A	September 11, 1990	N/A	000
N/A			
US 5047375 A	September 10, 1991	N/A	000
N/A			

APPLICATION-DATA:

PUB-NO	APPL-DESCRIPTOR	APPL-NO
APPL-DATE		
AU 8947058A	N/A	1989AU-0047058
December 21, 1989		
US 5047375A	N/A	1988US-0288681
December 22, 1988		

INT-CL (IPC): B03D001/02; C04B033/00 ; C09C001/42 ;  
C09C003/04 ;  
D21H003/82

ABSTRACTED-PUB-NO: AU 8947058A

BASIC-ABSTRACT: Iron stained TiO<sub>2</sub> contg. kaolin (I) is beneficiated esp. by froth flotation and froth rejects obtd. comprising kaolin enriched in iron stained TiO<sub>2</sub> are dewatered, dried and calcined at 1500-2200 deg. F to obtain pigment prod. (II). Starting material (I) is pref. a naturally occurring kaolin contg. 1-2 wt.% TiO<sub>2</sub>. Enriched kaolin contains e.g. 2-15% TiO<sub>2</sub>.

USE/ADVANTAGE - When used as a filler in paper, prod. (II)

imparts a brightness  
of 60-90 and paper has enhanced opacity compared to calcined  
kaolins not  
enriched in TiO<sub>2</sub>. (II) enhances puritability of newsprint grade  
paper.  
Retention of (II) in paper web is higher.

ABSTRACTED-PUB-NO: US 5047375A  
EQUIVALENT-ABSTRACTS: Prodn. of high opacifying pigment comprises  
subjecting an  
iron-stained titania-contg. kaolin to froth flotation, recovering  
froth  
rejects" which contain kaolin enriched in the iron-stained  
titania. Enriched  
kaolin contains 2-15 wt.% titania. Froth "rejects" are dewatered  
and dried.  
The rejects are then calcined at 1500-2200 deg.F. Pref. froth  
"rejects" are  
not further beneficiated prior to calcining. Esp. starting  
material is a  
naturally occurring kaolin contg. 1-2 wt.% titania.  
USE/ADVANTAGE - Kaolin  
pigment is useful as a filler in paper prods. Pigment has high  
opacity.

(5pp)

CHOSEN-DRAWING: Dwg.0/1

TITLE-TERMS:  
HIGH OPAQUE KAOLIN CONTAIN PIGMENT USEFUL FILL PAPER SHEET

DERWENT-CLASS: F09 G01 P41

CPI-CODES: F05-A06D; G01-A05; G01-A08; G01-A10;

SECONDARY-ACC-NO:  
CPI Secondary Accession Numbers: C1990-110149  
Non-CPI Secondary Accession Numbers: N1990-197128

DOCUMENT-IDENTIFIER: US 5168083 A  
TITLE: High opacity defined kaolin product and method of producing same

BSPR:

The present invention relates to a defined kaolin composition exhibiting high opacity and to a method of producing the kaolin composition by controllably defining a kaolin clay. More specifically, the present invention relates to a beneficiated kaolin product having improved opacifying efficiency thereby rendering the product functional as a high performance paper coating or filler, and to a method of producing the beneficiated kaolin product by controllably defining and otherwise treating a base kaolin clay so as to remove a substantial portion of colloidal particles therein.

BSPR:

It is well known in the paper industry, that a wide variety of pigments, such as titanium dioxide, calcium carbonate, talc, synthetic silicates, and clays such as bentonite and kaolin, are suitable for use as paper fillers and/or coatings. Kaolin, a naturally occurring hydrated aluminum silicate, is presently the most widely utilized and is available in a range of particle sizes and brightnesses, as well as being either delaminated or non-delaminated. Hydrated kaolin is white in color, has a fine particle size, is relatively chemically inert, and makes an ideal low cost paper filler. Although calcined (anhydrous) kaolin is also available for use as a paper filler and can impart greater opacity to paper than the hydrated kaolin, it has the serious disadvantage of being more abrasive.

BSPR:

Prior art kaolin paper fillers and coatings are typically produced by a beneficiation process which typically consists of fractionating